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DESIGN-TECHNOLOGY INSTITUTE OF HIGH RATE HYDRODYNAMICS

THE PRELIMINARY STUDIES OF SURVIVABILITY OF NOZZLE THROAT MATERIALS IN ULTRAHIGH PRESSURE OF NITROGEN AND AIR

(The final report on the work performed
under the contract No. F61708-97-W-0138)

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13. ABSTRACT (Maximum 200 words)

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1. INTRODUCTION.

One of the most complicated problems of the up-to date experimental aerodynamics in simulation a hypersonic flight is the survival of a nozzle throat of a wind tunnel. An attempt to produce a flow ahead of a model, provided that the flow parameters are closely approximating natural those by general similarity criteria, leads to a rise of gas parameters in a settling chamber. In the modern operating facilities the pressure is up to 1 *GPa* [1], temperatures are from 2000 to 10000 *K* [2] for an operating time from a millisecond to several tenth fractions of a second.

With such stagnation parameters, the nozzle throat is subjected to powerful force, temperature and corrosion action of the flowing out air. When exhausting a gas its pressure to a nozzle wall varies along the flow direction from values, which are practically equal to the pressure in the settling chamber, to those approximating the atmosphere pressure and lower than it. This causes the occurrence of a complicated stressed state and the necessity of providing the external support of a nozzle insert in order for the state to approximate, as much as possible, that resembles the all-round compression. In this manner there arises a limitation of the survivability caused by the material strength. Our experience suggests that if the material strength is inadequate to the given pressure in the settling chamber, the plastic strains will cause the material flowing over and the nozzle throat shutting off. As we shall see later, this effect is most pronounced if the standard or oxide-alloyed copper is used as a nozzle insert.

Stresses caused by a high pressure are applied with thermal stresses resulted from the warming-up of the inner surface of the nozzle and the corresponding thermal expansion of the material. This is reflected in increase forces rupturing the insertion from the inside, as viewed from the through-hole.

Alternatively, the warming-up of the inner layer of the insert leads to reduce the material strength and it begins "to be taken out" from the nozzle by tangential forces caused by wall friction of a gas. Here, the increase of the throat diameter is observed. Such examples will be likewise seen.

The high oxidation power of the air may have the more pronounced and dangerous effect. Suffice it to say that

when increasing the pressure up to 1 *GPa* even at constant temperatures, the oxidizing reactions increase about by 10000 times in velocity! Actually the materials which are non-persistent to oxidation under these conditions simply burn in contact with the airflow. As indicated in one of our experiments, a flame therewith may spread over adjacent details, which are out of such a thermal-stressed state and have remained intact under multiple similar exposure.

From the above discussion it is apparent that the insertion material has to possess a unique combination of properties, such as a maximum high strength, a thermal shock resistance and an oxidizing action of air.

The availability of the A-1 gasdynamic adiabatic compression facility capable of producing a gas pressure up to 1 *GPa* at a temperature of 2000 *K* in the Lavrentyev Institute of Hydrodynamics has made it possible to perform a preliminary research on the survivability of various materials in the nozzle throat. The results of the research are given in the present report. According to the contract name, the results should be considered as tentative because more comprehensive and complete researches are need far time consuming and call for larger scope of financing.

From the A-1 facility specificity, all experiments have been basically performed given parameters lying close to an adiabat passing through the pressure point of about 1 *MPa* at room temperature.

2.MATERIALS

2.1.ALLOYED COPPER

Internally oxidized copper (IOC) is a dispersively reinforced alloy Cu+3,5% vol. of Al_2O_3 manufactured by the powder metallurgy method. A powder or cuttings made from the alloy Cu+0,4% of Al have been internally oxidized as Al has been turned to Al_2O_3. On oxidation, the material presents a pure copper matrix with distributed Al-oxide particles of which size is not over 30 *nm*. A briquette having an open porous density has been made from a powder fraction by the multi-stage method in the pressurizing regime at room temperature.

A rod having a stretch coefficient μ, which is equal to 10, has been extruded from the briquette heated to 1000 *C* with the help of high-frequency currents. To produce a thin structure and, consequently, an added strengthening, the above rod has been re-extruded without heating for μ=3.7. Thereafter all treated rods have a very textured structure involving practically non-etched boundaries of particles, fractions and grains. Test samples have been produced from the rod.

Internally oxidized alloys differ from copper bronzes by the stable structure to temperatures near that of the copper melting [3]. This determines the conservation of their strength properties at high temperatures. Figure 1 of [3] gives the plots of the dependence of the hardness (by Rockwell, F-scale) of the pure riveted on 50 percent of copper and the above alloy on the annealing temperature. One can see that the alloy retains its properties up to the copper melting temperature while the pure copper loses its hardness practically fully at temperatures less than 400 C.

Figure 2 of the same paper gives the dependence of the long time (100 hours) strength of copper, stainless steel 304, the Monel alloy (70% Ni+30% Cu) and the alloy Cu+3,5 vol.%Al_2O_3. As one can see, at the temperature more than 670 C the alloy becomes better in magnitude of the long time strength limit than steel.

In the course of loading material exists in a complex stress state. In addition to compression the layers near the hole wall are subjected to high shearing stresses. Even so, after 15 cycles of loading the investigations with help of optical microscope have revealed that the IOC structure is unaffected (fig. 3a). Intragrain cracks and pores are not observed. The orifice change observed is due

to concurrent processes: the carrying away of a material by gas flow and strains owing to complex force and temperature stresses.

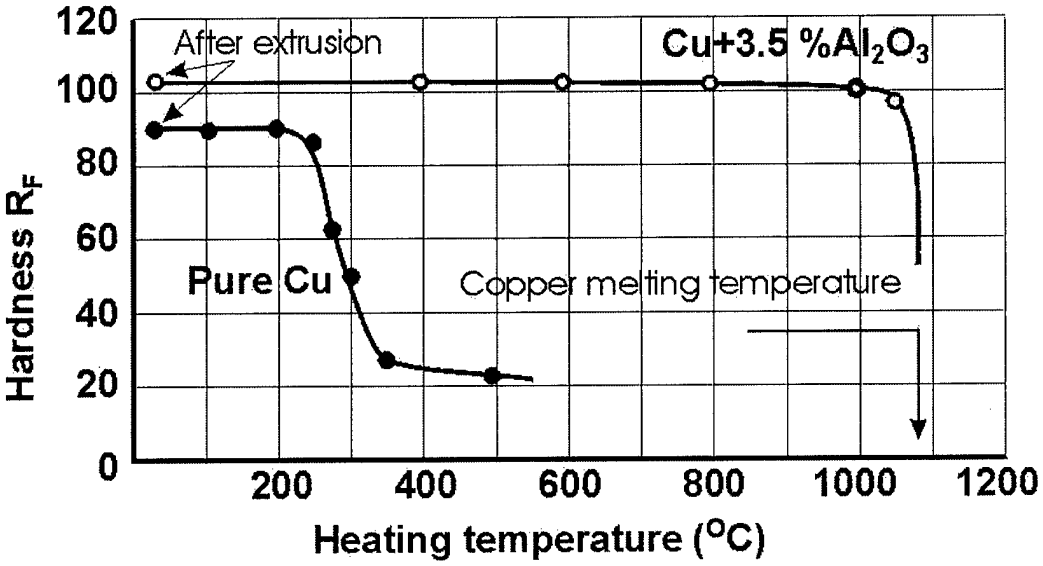

Fig.1.

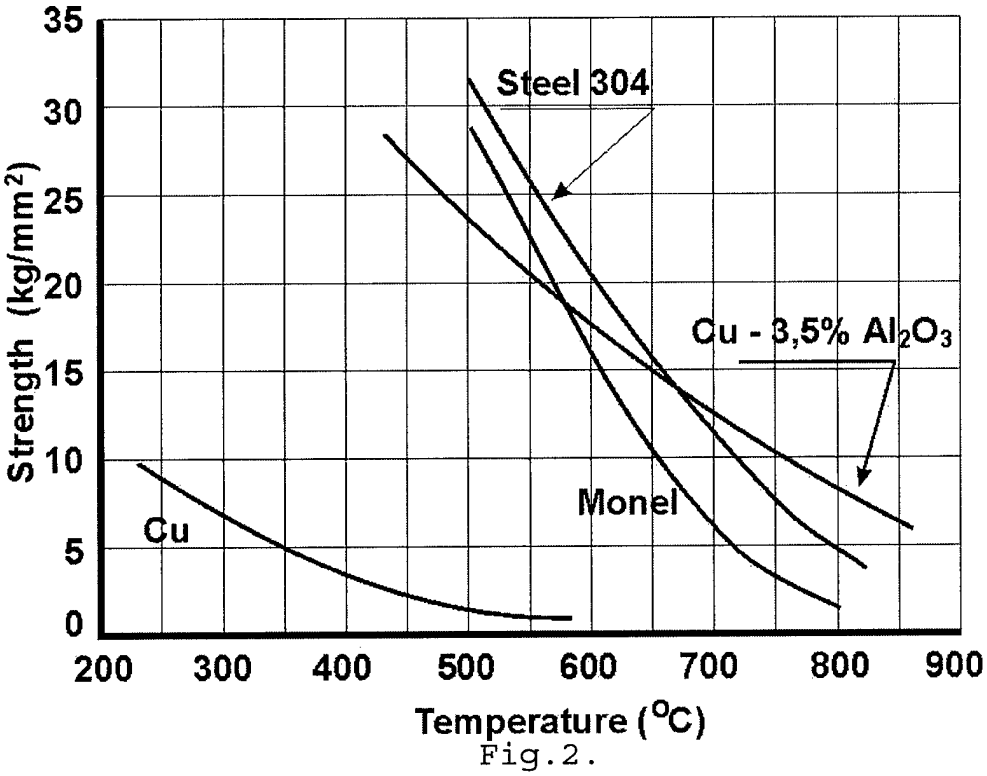

Fig.2.

When producing the material, including the internal oxidation process, the severely fragmented and textured IOC structure has no energy-isolated local volume sites

a

b

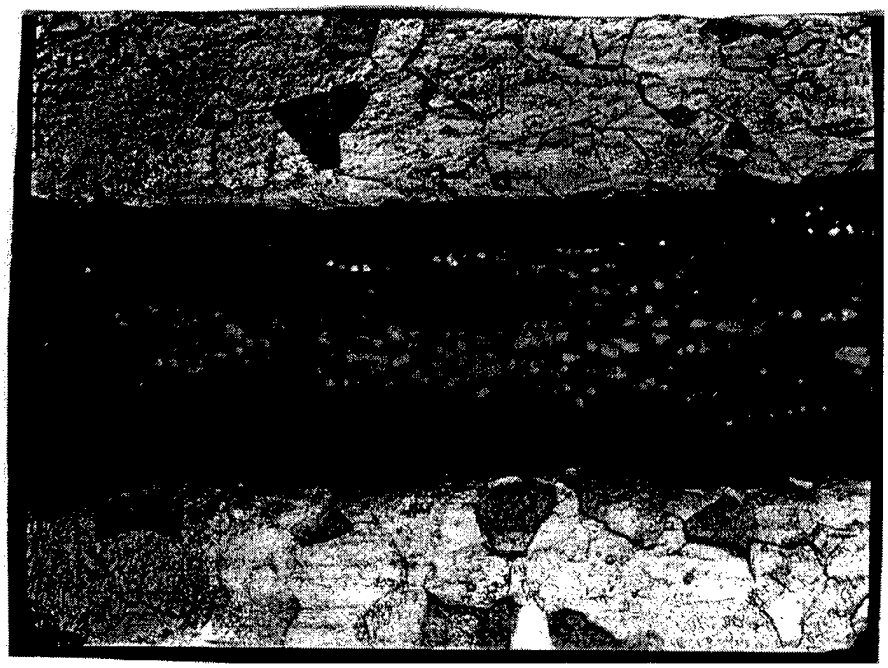

Fig.3

where microcracks transformed into ribs for removal of a material in the form of flakes might originate.

2.2.TANTALUM

A fabricated nozzle insert made of tantalum has been presented by a collaborator of the Livermore National Laboratory Dr. M.Costantino. As revealed in our metal-

lographic study, the tantalum structure in its original state is characterized by an equiaxiality of grains, whose mean size is equal to 150 *nm*.

Ribs, their localization and forms visible in the tested sample of tantalum (fig.3*b*) testify to the fact that they originate at grain boundaries located at an acute angle to the shear stress direction.

The findings of our investigations indicate that tantalum behavior in applied loading conditions is suited to the well-known conception of that at high temperatures the weakest points are grain boundaries, i.e. positions having a high density of defects and hence a high initial energy. Unlike tantalum, the IOC structure severely dispersed and textured while in production of a material, including the process of the internal oxidation has no energetically isolated local volume sites where microcracks transformed into ribs of a material removable in the form of flakes might originate.

Thus, the wearing mechanism in applied loading conditions is determined to a greater extent by the structure state of an initial material.

2.3. RHENIUM

As well as tantalum, the Livermore National Laboratory has presented a finished nozzle insert made of rhenium. After slightly fitting of an outer size the insert was set to an iron ring and fixed at the output of the settling chamber.

3. NOZZLE INSERTS, LOADING CONDITIONS, EXPERIMENTAL FINDINGS.

3.1 NOZZLE INSERTS.

A drawing for a tested nozzle insert is given in Fig.4. The outer sizes and angles of conical surfaces in all experiments are the same, exclusive of WC. The diameter of the critical throat section, which before testing ranges from 0,28 to 0,638 mm, will vary from sample to sample or from experiment to experiment.

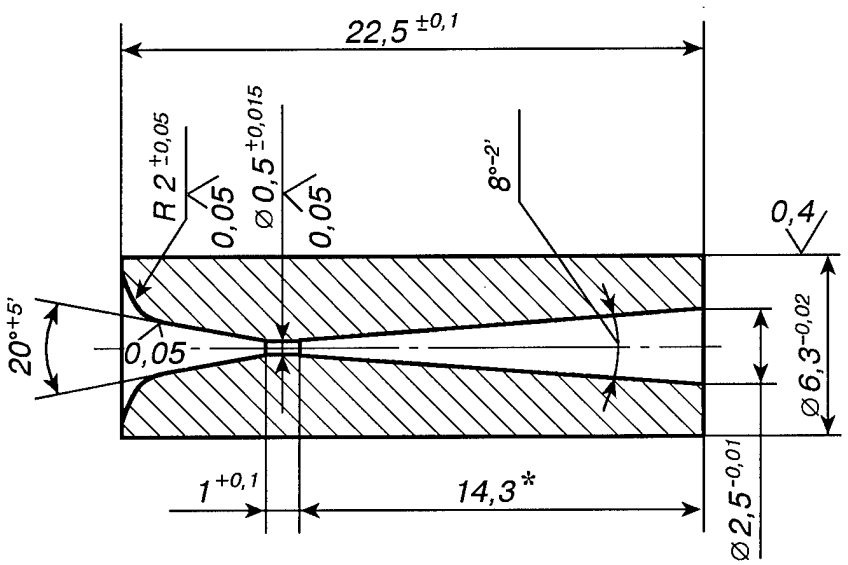

Fig.4.

3.2.HEAT LOADS.

Before proceeding to the findings, it is necessary to estimate the temperature values to which the insert surface is heated during experiments. To do this requires the solution of the heat-transfer equation for an infinite space at which boundary there arises a heat exchange with the environment (a boundary condition of the third type: $-\frac{\partial\theta}{\partial x}+h\theta=0$, for $x=0$ [4], with the initial constraint: $\theta(x)=0$, at $t=0$.

The solution has the following form:

$$\theta=\Phi^*\left(\frac{x}{2\sqrt{\kappa t}}\right)-\left[\Phi^*\left(\frac{x}{2\sqrt{\kappa t}}+h\sqrt{\kappa t}\right)\right]\exp\left\{hx+h^2\kappa t\right., \qquad (1)$$

where $\Phi^*(\xi)=1-\mathrm{erf}(\xi)$, $\theta=(T-T_n)/(T_0-T_n)$, κ - is the thermal conductivity, t is the time from the start of heating, x

is the coordinate measured from a heated inner surface of the insert; T_n and T_o are the initial temperature of a body and the gas stagnation temperature, respectively; λ - the heat conductivity, $\kappa=\lambda/c\rho$ is the thermal conductivity coefficient of the insert material, $1/h=\kappa/\lambda=x_0$ is the intrinsic length. At the inner surface of the insert $(x=0)$:

$$\theta_W = 1 - \Phi^*\left(h\sqrt{\kappa t}\right)\cdot \exp\left\{h^2\kappa t\right. , \qquad (2)$$

and the surface temperature is determined by the formula:

$$T_W = \theta_W T_0 + (1-\theta_W)T_n .$$

The value of $1/h^2\kappa = t_0$ is the characteristic time consistent with θ_W = 0.5724.

The key thermophysical property and the ultimate strength of materials which can, in some way or other, be used for manufacturing of the nozzle inserts or in solving of a problem of a raise of their survival are given in Table 1. Basic information source is the fundamental manual []. As it is visible from this table, for all given materials t_0 lies in limits from 4.5 up to 0.1 *ms*, and x_0 between 0,8 and 0,03 *mm*

The Table 2 gives the conditions and results of the experiments performed with different materials. In addition to the pressure and the temperature in the settling chamber and the test time, in the table are given non-dimensional time ($h\sqrt{\kappa t}$) and wall temperature (θ_W) parameters, that are heed for estimation of wall surface temperature (T_W). Stars point experiments with using of air.

In the same place the temperature (T_W), to which on the mentioned above evaluation surface of a throat was heated up, depth of a warm-up to temperature 0,9 T_W, and also diameters of a throat of the nozzle before shots and their change after experiment are given. The throat diameter measurements are taken by special wire gauges with an accuracy of no worse than 0,002 *mm*.

In all calculations the value of the heat transfer coefficient (α) has been taken as equal to 55 W/cm/K. The value has been obtained by semiempirical estimations on evidence of different authors and confirmed by experiments with heat losses measured in the A-1 facility performed previously by authors of the report [ ].

The temperatures given in the table appear must be considered as somewhat overestimated because in calculati-

Table 1.The thermophysics properties and strength of used and some other materials.

Material	Molecul mass μ ($g/mole$	Dens. (ρ) (g/cm^3)	Heat cap. C ($J/g/K$)	eat con duct. λ ($W/cm/K$)	Thermal conduct. (cm^2/sec	Char. time $t_0=\rho\lambda C/\alpha^2$ ($msec$)	Charact. size $x_0=1/h$(cm	Melting tempera ture. (K	Ult.re-sist σ_{ur} (N/m^2) :10^7
Copper	63.5	8.9	0.386	3.89	1.132	4.418	0.0707	1356	20-25
Tantalum	181.0	16.6	0.14	0.75	0.323	0.576	0.0136	3269	31-45
Rhenium	186.2	20.5	0.136	0.5	0.179	0.461	0.0091	3453	108
Platinum	195.1	21.4	0.132	0.82	0.290	0.766	0.0149	2042	14
Iridium	192.2	22.4	0.13	1.38	0.474	1.328	0.0251	2716	23
7Pt+3Ir	194.2	21.6	0.132	0.309	0.108	0.291	0.0056	-	143-116
Or	197.0	19.3	0.128	3.1	1.255	2.532	0.0564	1336	12.4
Silver	107.9	10.5	0.236	4.18	1.687	3.424	0.0760	1234	13.5
Sapphire	102.0	4.0	0.758	0.167	0.055	0.167	0.0030	2313	200/70
Tungsten	183.9	18.8	0.135	1.15	0.453	0.965	0.0209	3653	70-81
Molybden	95.9	9.0	0.248	1.31	0.587	0.967	0.0238	2898	30.8
(diam)	12.0	3.52	0.51	1.34	0.746	0.795	0.0244	-	-
Steel	$\approx$56	7.87	0.46	0.3	0.083	0.359	0.0055	1670	150-10
WC-6	187.8	15.6	0.192	0.291	0.097	0.289	0.0053	2993	194-163

Table 2. The conditions and results of the experimentsand esimation findings.

	Material	ax. plen press, P_{max} (*MPa*	ax, plen, temperat. T_0 (*K*)	Test time (*msec*)	Char. time t_0 (*msec*	$=\sqrt{t/t_0}$	θ_w	Wall urf.,temp rat. T_w (*K*	$x=\sqrt{Kt}$ (*mm*)	Throat dia, before exp. *d* (*mm*)	Δd (*mm*)
1		***541.6**	**1592**	**280**		**7.961**	**0.930**	**1501**	**1.126**	**0.28**	**0**
2	Cu	656.6	1667	250	4.418	7.522	0.926	1565	1.064	0.28	0
3	+3%Al_2O_3	435.4	1515	30		2.606	0.796	1266	0.369	0.28	0
4		760.0	1731	35		2.815	0.810	1457	0.398	0.28	-0.6
5		598.0	1631	68		9.422	0.940	1551	0.221	0.545	0
6		816.0	1761	56		8.550	0.934	1665	0.200	0.545	0.04
7		407.1	1483	60		8.850	0.937	1408	0.207	0.585	0.004
8		608.0	1700	30		6.258	0.911	1575	0.147	0.589	0.031
9		434.0	1532	50		8.079	0.931	1446	0.189	0.620	0
10		576.5	1613	42		7.405	0.924	1513	0.173	0.620	0
11	Renium	540.0	1614	40	0.766	7.226	0.923	1512	0.169	0.620	0
12		621.7	1671	50		8.079	0.931	1575	0.189	0.620	0.002
13		664.0	1718	35		6.760	0.917	1600	0.158	0.622	0.006
14		427.6	1525	42		7.405	0.924	1432	0.173	0.635	0
15		583.0	1641	40		7.226	0.923	1537	0.169	0.635	0.003
16		706.5	1695	36		6.855	0.918	1581	0.200	0.638	0.012
17		***389.6**	**1433**	**56**		**8.550**	**0.934**	**1358**	0.200	**0.650**	**0.005**
18		***481.0**	**1510**	**44**		**7.580**	**0.926**	**1420**	0.177	**0.655**	**0.006**
19	Tanalum	467.0	1545	110	0.576	13.82	0.959	1494	0.377	0.515	-0.05
20	Sapphir	515,0	1600	50	0.167	17.30	0.967	1557	0.105	0.573	0

*The tests with air.

ons the stream temperature has been taken as equal to that of stagnation without considering a reduction coefficient and losses in a gas-dynamic channel.

The first conclusion, which follows from this table, is that as the parameter τ (with the exception of short-time experiments with copper (line 3 and 4)) considerably exceeds unit, the surface of the tested materials got warm more than up to 90% of stagnation temperature.

Second - the thickness of a layer which is warmed up to temperature about 0,9 T_w, for a rhenium does not exceed 0,22 *mm*, for sapphire it is 0,1 *mm*.

Since the heated layer depth is considerably less than the nozzle throat diameter, the above solution describes process of heating rather well. Even though, all other factors being equal, it provides a slightly higher temperature of the surface both owing to the geometric reasons, and owing to losses of an enthalpy at driving of gas on a tract of facility. However, here, an error will not exceed the value caused by inaccuracy in defining of heat-transfer coefficients, and by the influence of the temperature dependence of physical parameters of a material.

2.2.FORCE EFFECT.

As an illustration of the force effect character, in figure 5 is shown a real pressure oscillogram obtained in one of our experiments. One can easily observe the first rise up to the level about 150 *MPa* caused by the operation of the A-1 first stage. Then the second stage raises the pressure to the value, at which the test of an insert will be carried out and displaces gas from a settling chamber.

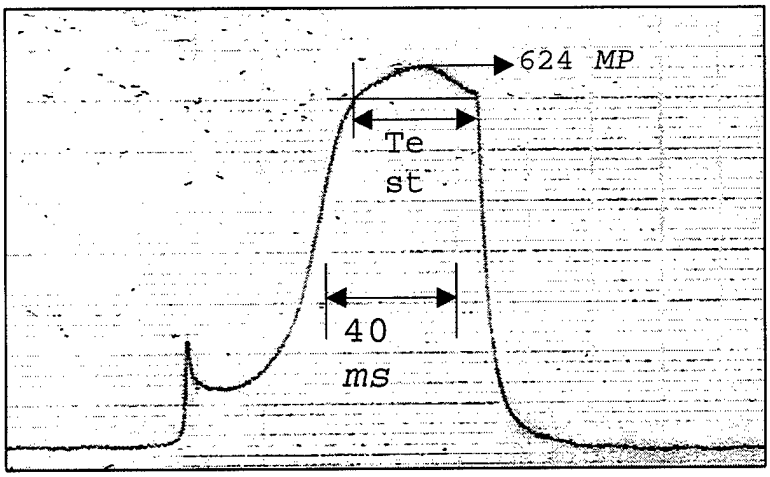

Fig.5

The pressure on an interior surface of a nozzle insert varies according to the gas dynamics laws (in view of the equation of state) from the value equal to the pressure of the settling chamber (P_o) at its far left part (fig.4), approximately to 0,4 P_o in the nozzle throat and then down to about 0,003 P_o at the nozzle insert end. Outside of an insertion to protect it from a rupture the uniformly distributed supporting pressure equal to that of the settling chamber acts. One can see that the nozzle throat is in a rather complicated stressed state and the inner layers are axially affected by high shear stresses due to the lack of support from the supersonic part of the nozzle.

Therefore, once a pressure at which the material as a whole ceases to counter the operating forces is attained, the insert is crumpled, the material is forced into the hole and the latter is shut down. Figure 6 illustrates a photo of a sectional view of the copper insert acted upon by pressure such as 600 *MPa*. Experiments with the alloyed copper have shown that its strength restriction approach at a pressure of about 760 *MPa*, and in this case there is no catastrophic change as in Fig.6: the throat diameter is reduced only to 60 *μm*. However, the reduction of the nozzle end diameter (from 2,6 to 2 mm) have been observed.

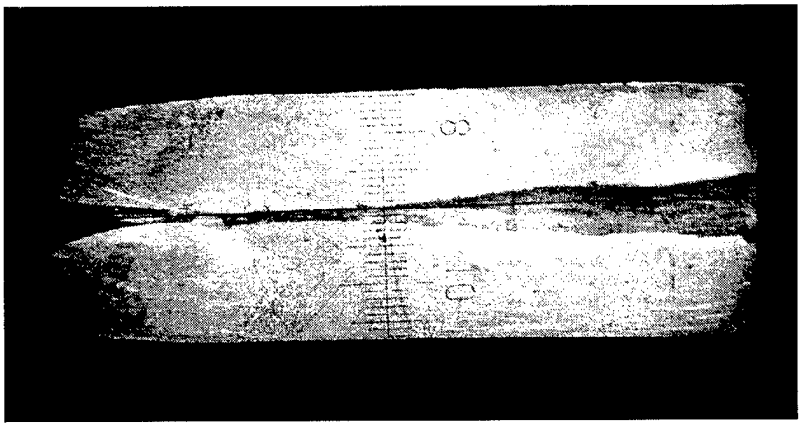

Fig.6.

2.3. CORROSION EFFECT

Used in experiments with nitrogen is a gas obtained by evaporating liquid technical nitrogen in a special evaporator. This nitrogen contains always an admixture of oxygen whose concentration increases several thousand times with increasing pressure. In spite of this, no noticeable erosive action has been observed in our experi-

ments with nitrogen, and while the pressure did not become rather high (300 *P*), for relatively low-strength materials (Cu, Ta, Cu+3,5%Al_2O_3), the throat diameter remained constant (within the limits of measurement error), or the tendency to its diminution was observed.

As to a strength material like rhenium, at pressures below 600 *MPa* there are no changes in the throat diameter at all. However, a slow (from 2 to 10 *μm* per experiment) rise of diameter is observed under pressures from 600 to 700 *MPa* and at 816 *MPa* the change measures 40 *μm* per diameter (Table 2).

Attention is drawn to the fact that the hole retains well its round form in all materials given minor changes of the diameter.

Diagram in fig.7 illustrates a rupturing process of a draw plate simulating a nozzle throat of diameter 0,35 mm made from tungsten carbide doped with 6% cobalt. Numbers just over the column point to the nitrogen pressure values such that the tests were performed. One can see the throat diameter stabilization after three first shots, however at the tenth shot the catastrophic failure arises under a pressure below that of two previous tests. This suggests that a progressive accumulation of material defects takes place under the flow action, which terminates in failure.

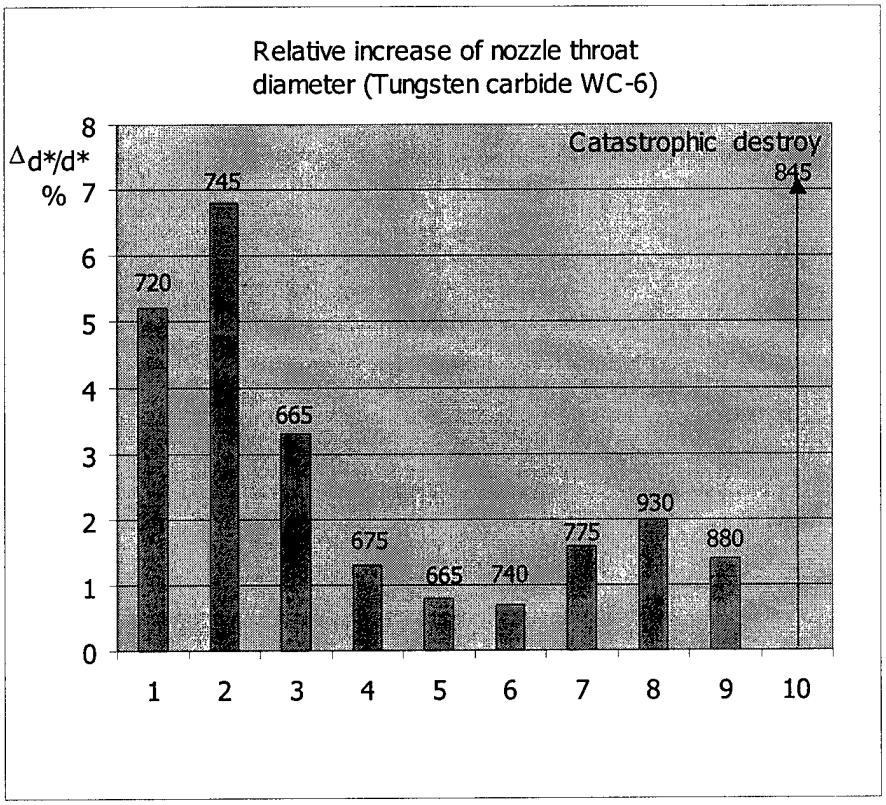

Fig.7.

The main reason of such defects occurring is thermal stresses caused by non-uniform heating of a material. The surface layer of the throat heats up during the flow out to the high temperature, whereas the whole remaining body of the insert is left cold (see above). As the pressure drops in the settling chamber and the outside support disappears, the insert body is broken by these stresses.

This is most evidenced by the example of the sapphire insert whose body, as found post-experimentally, has been covered by cracks. All cracks have outgone from the nozzle throat and in this case the outer and inner sizes of the insert have remained invariable. The test conditions are P_o=515 *MPa*, T_o=1600 *K*, the exposure time is 50 *ms*.

A completely different picture of material fracture occurs on interacting with air. Inserts made from tungsten and iron-bearing alloys fail accidentally at pressures above 400 *MPa* even in the first start. The failure of the insert made from the VASCO SUPREME alloy is exemplified in Fig.8. In this experiment the pressure consisted of 500 *MPa* at the temperature of about 1600 *K*. Under these conditions, all inserts made of alloyed copper are as yet well efficient. The reason of this fast failure appears to be the joint action of the high temperature and the oxygen concentration, which causes simply the metal ignition and the insert burning out.

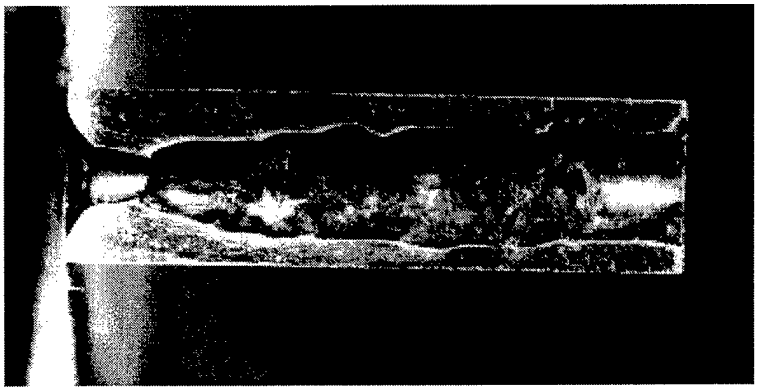

Fig.8.

DISCUTION AND CONCLUSIONS.

1. As indicated in present preliminary studies, rhenium is the most promising material for the nozzle throat as a technical nitrogen is operated under pressures about 700 MPa. Copper alloyed with 3,5% aluminum oxide and manufactured by a special technology sustains pressures only slightly lower than 650 *MPa*.

2.A comparison between shears of this copper and, say, tantalum shows that the most probable reason of retaining the high strength of the alloyed copper, practically up to the melting temperature, is its microstructure, which makes dislocation displacements difficult. A comparison of lines 2 and 4 in Table 2 shows clearly that for the oxide-strengthened copper the limitation of practical applicability is primarily due to its strength properties because the nozzle strains have been found at a pressure of 760 *MPa* when an estimated wall temperature has been by 112 *K* below than at 656,6 *MPa*.

3.The most promising materials in operations with air may be inserts made of alloyed copper (up to pressure about 650-700 *MPa*), and high melting oxides of aluminum or zirconium in forms, which are provided with the maximum possible temperature conductivity. If these materials are used it is essential to have the outside force protection guaranteeing the lack of tensile stresses in the insert body even with heating its inner layer.

4.Our experience on exploitation of the A-1 facility and experiments performed in the framework of the present contract show that the most high-strength alloys containing iron and tungsten at the stagnation temperature above 900 *K* can be used both with air and with technical nitrogen only providing the inner surface of the nozzle will be protected from direct contacts with gas by an oxidation-resistant cover, such as by platinizing.

5. As the contract name suggests, the studies conducted are preliminary and reveal such material types, which should be further investigated regularly. Within the limits of assigned means and time, we have tried to do our best despite all difficulties and emergencies arisen through no our fault.

6. In the further researches, which we assume to continue, it is necessary to conduct systematical tests of insert made from sapphire, rhenium, and alloyed copper in more broad range of temperatures and pressure. In addition, it is essential to be taken by development of protective coatings technology for the strongest materials.

Otherwise the exit on pressures more than 1000 will be hardly possible.

It would be good if EOARD could support this further investigation!

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